



(51) International Patent Classification:

*D01F 8/16* (2006.01)      *B01D 39/16* (2006.01)  
*D01F 8/06* (2006.01)      *D01D 5/30* (2006.01)

(21) International Application Number:

PCT/US2014/033246

(22) International Filing Date:

8 April 2014 (08.04.2014)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

13/860,076      10 April 2013 (10.04.2013)      US

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(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

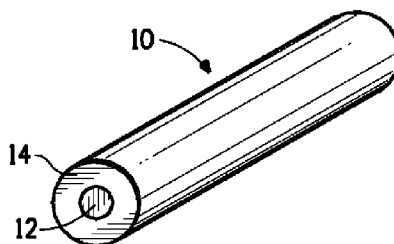
(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Published:

— with international search report (Art. 21(3))

(54) Title: ACID RESISTANT FIBERS OF POLYARYLENE AND POLYMETHYLPENTENE

FIG. 1



(57) Abstract: A multicomponent fiber having an exposed outer surface with the fiber having at least a first component of polyarylene sulfide polymer; and at least a second component of a thermoplastic polymer free of polyarylene sulfide polymer, wherein said thermoplastic polymer forms the entire exposed surface of the multicomponent fiber and is polymethylpentene.



## **TITLE**

Acid Resistant Fibers.of Polyarylene and Polymethylpentene

### **BACKGROUND OF THE INVENTION**

#### **1. Field of the Invention**

The present invention relates to fibers having a polyarylene sulfide component and products including the same.

#### **2. Description of the Related Art**

Filtration processes are used to separate compounds of one phase from a fluid stream of another phase by passing the fluid stream through filtration media, which traps the entrained or suspended matter. The fluid stream may be either a liquid stream containing a solid particulate or a gas stream containing a liquid or solid aerosol.

For example, filters are used in collecting dust emitted from incinerators, coal fired boilers, metal melting furnaces and the like. Such filters are referred to generally as "bag filters." Because exhaust gas temperatures can be high, bag filters used to collect hot dust emitted from these and similar devices are required to be heat resistant. Bag filters can also be used in chemically corrosive environments. Thus, dust collection environments can also require a filter bag made of materials that exhibit chemical resistance. Examples of common filtration media include fabrics formed of aramid fibers, polyimide fibers, fluorine fibers and glass fibers.

Polyphenylene sulfide (PPS) polymers exhibit thermal and chemical resistance. As such, PPS polymers can be useful in various applications. For example, PPS can be useful in the manufacture of molded components for automobiles, electrical and electronic devices, industrial/mechanical products, consumer products, and the like PPS has also been proposed for use as fibers for filtration media, flame resistant articles, and high performance composites.

Despite the advantages of the polymer, however, there are difficulties associated with the use of fibers from PPS because PPS has limited resistance to extremely acid environments.

What is needed is a fiber that combines the high temperature properties of PPS that can be used in acidic environments.

### **SUMMARY OF THE INVENTION**

In one embodiment, the present invention is directed to a multicomponent fiber having an exposed outer surface, comprising: at least a first component of polyarylene sulfide polymer; and at least a second component of a thermoplastic polymer free of polyarylene sulfide polymer, wherein said thermoplastic polymer forms the entire exposed surface of the multicomponent fiber and consists essentially of polymethylpentene.

The invention is further directed to a method for increasing the acid resistance of any of the embodiments of a polyarylene fiber described herein by providing it with a coating of the second component in any of the embodiments described herein.

In particular the method for improving the acid resistance of a fiber comprises the steps of;

- i. providing a fiber,
- ii. coating the fiber with a thermoplastic polymer that is free of polyarylene sulfide polymer to form a coated fiber, wherein said thermoplastic polymer forms the entire exposed surface of the coated fiber and consists essentially of a polymethyl pentene,

said fiber comprising: at least a first component of polyarylene sulfide polymer.

### **BRIEF DESCRIPTION OF THE FIGURES**

Figure 1 is a transverse cross sectional view of an exemplary fiber configuration useful in the present invention.

Figure 2 illustrates a cross sectional view of an islands-in-the-sea fiber.

Figure 3 illustrates an embodiment with multilobal structure.

### **DETAILED DESCRIPTION OF THE INVENTION**

Applicants specifically incorporate the entire contents of all cited references in this disclosure. Further, when an amount, concentration, or other value or parameter is given as either a range, preferred range, or a list of upper preferable values and lower preferable values, this is to be understood as specifically disclosing all ranges formed from any pair of any upper range limit or preferred value and any lower range limit or preferred value, regardless of whether ranges are separately disclosed. Where a range of numerical values is recited herein, unless otherwise stated, the range is intended to include the endpoints thereof, and all integers and fractions within the range. It is not intended that the scope of the invention be limited to the specific values recited when defining a range.

For purposes of illustration only, the present invention will generally be described in terms of a bicomponent fiber comprising two components. However, it should be understood that the scope of the present invention is meant to include fibers with two or more structured components.

In one embodiment the invention is directed to a multicomponent fiber having an exposed outer surface. The fiber comprises: at least a first component of polyarylene sulfide polymer; and at least a second component of a thermoplastic polymer free of polyarylene sulfide polymer, wherein said

thermoplastic polymer forms the entire exposed surface of the multicomponent fiber. The second component consists essentially of polymethylpentene (PMP.) By "consists essentially of" is meant that any additional component does not detract from the performance of the structure.

The fiber of the invention has an unexpectedly high resistance to acid compared to polyarylene sulfide alone, and this result is unexpected because PMP is recognized as having a high permeability, as evidenced by its permeability to oxygen. (Data supplied by the manufacturer, Mitsui Chemicals showing the permeability of PMP.)

The polyarylene sulfide polymer may comprise in one embodiment a polymer in which at least 85 mol % of the sulfide linkages are attached directly to two aromatic rings.

In a further embodiment the polyarylene sulfide polymer is polyphenylene sulfide.

The second component may be present at a 10 to 30% by weight of the total polyarylene sulfide plus thermoplastic polymer. In a further embodiment the second component may comprise less than about 30 percent by weight or even 20% by weight of the total weight of the fiber.

The fiber may be a continuous filament or a staple fiber. It may also be a spunbond fiber or a meltblown fiber.

The fiber may be a bicomponent fiber comprising a sheath component and a core component, wherein said sheath component forms the entire exposed outer surface of said fiber and comprises said thermoplastic polymer free of polyarylene sulfide polymer, and wherein said core component comprises polyarylene sulfide polymer. In a further embodiment the bicomponent fiber has a concentric sheath/core cross section. In a still further embodiment the bicomponent fiber has an eccentric sheath/core cross section.

The fiber may be an islands-in-the-sea fiber comprising a sea component and a plurality of island components distributed within said sea component,

wherein said sea component forms the entire exposed outer surface of said fiber and comprises said thermoplastic polymer free of polyarylene sulfide polymer, and wherein said plurality of island components comprises polyarylene sulfide polymer.

The invention is also directed to a web comprising the fiber of any of the embodiments described above. The web may comprise a woven or nonwoven material. The web may also be made by a spunbond or meltblown process.

Turning now to the figures, Fig. 1 is a transverse cross sectional view of an exemplary fiber configuration useful in the present invention. Fig. 1 illustrates a bicomponent fiber **10** having an inner core polymer domain **12** and surrounding sheath polymer domain **14**. Sheath component **14** is formed of a thermoplastic polymer free of polyarylene sulfide polymer. Core component **12** is formed of polyarylene sulfide polymer. In the present invention, sheath **14** is continuous, e.g., completely surrounds core **12** and forms the entire outer surface of fiber **10**. Core **12** can be concentric, as illustrated in Fig. 1. Alternatively, the core can be eccentric, as described in more detail below. Also, it should be recognized that due to processing variability, a small portion of the sheath could be contacted by the polyarylene sulfide polymer, however it is believed that there would only be minimal effect on spinning ability. Regardless, the sheath should be as virtually free of polyarylene sulfide polymer.

Other structured fiber configurations as known in the art can also be used, so long as the thermoplastic polymer free of polyarylene sulfide polymer forms the entire exposed outer surface of the fiber. As an example, another suitable multicomponent fiber construction includes "islands-in-the-sea" arrangements. Fig. 2 illustrates a cross sectional view of one such islands-in-the-sea fiber **20**. Generally islands-in-the-sea fibers include a "sea" polymer component **22** surrounding a plurality of "island" polymer components **24**. The island components can be substantially uniformly arranged within the matrix of sea component **22**, such as illustrated in FIG. 2. Alternatively, the island components can be randomly distributed within the sea matrix.

Sea component **22** forms the entire outer exposed surface of the fiber and is formed of a thermoplastic polymer free of polyarylene sulfide polymer. As with core component **12** of sheath core bicomponent fiber **10**, island components **24** are formed of polyarylene sulfide polymer. The islands-in-the-sea fiber can optionally also include a core **26**, which can be concentric as illustrated or eccentric as described below. When present, core **26** is formed of any suitable fiber-forming polymer.

The fibers of the invention also include multilobal fibers having three or more arms or lobes extending outwardly from a central portion thereof. FIG. 3 is a cross sectional view of an exemplary multilobal fiber **30** of the invention. Fiber **30** includes a central core **32** and arms or lobes **34** extending outwardly therefrom. The arms or lobes **34** are formed of a thermoplastic polymer free of polyarylene sulfide polymer and central core **32** is formed of polyarylene sulfide polymer. Although illustrated in Fig. 3 as a centrally located core, the core can be eccentric.

Any of these or other multicomponent fiber constructions may be used, so long as the entire exposed outer surface of the fiber is formed of the thermoplastic polymer free of polyarylene sulfide polymer.

The cross section of the fiber is preferably circular, since the equipment typically used in the production of synthetic fibers normally produces fibers with a substantially circular cross section. In bicomponent fibers having a circular cross section, the configuration of the first and second components can be either concentric or acentric, the latter configuration sometimes being known as a "modified side-by-side" or an "eccentric" multicomponent fiber.

Advantageously, the sheath/core fibers of the invention are concentric fibers, and as such will generally be non-self crimping or non-latently crimpable fibers. The concentric configuration is characterized by the sheath component having a substantially uniform thickness, such that the core component lies approximately in the center of the fiber, such as illustrated in Fig. 1. This is in contrast to an eccentric configuration, in which the thickness of the sheath

component varies, and the core component therefore does not lie in the center of the fiber. Concentric sheath/core fibers can be defined as fibers in which the center of the core component is biased by no more than about 0 to about 20 percent, preferably no more than about 0 to about 10 percent, based on the diameter of the sheath/core bicomponent fiber, from the center of the sheath component.

Islands-in-the-sea and multi-lobal fibers of the invention can also include a concentric core component substantially centrally positioned within the fiber structure, such as cores **26** and **32** illustrated in Figs. 2 and 3, respectively. Alternatively, the additional polymeric components can be eccentrically located so that the thickness of the surrounding thermoplastic polymer free of polyarylene sulfide polymer component varies across the cross section of the fiber.

Any of the additional polymeric components can have a substantially circular cross section, such as components **12**, **24** and **32** illustrated in Figs. 1, 2 and 3, respectively. Alternatively, any of the additional polymeric components of the fibers of the invention can have a non-circular cross section.

Polyarylene sulfides include linear, branched or cross linked polymers that include arylene sulfide units. Polyarylene sulfide polymers and their synthesis are known in the art and such polymers are commercially available.

Exemplary polyarylene sulfides useful in the invention include polyarylene thioethers containing repeat units of the formula  $—[(Ar^1)_n—X]_m—[(Ar^2)_i—Y]_j—(Ar^3)_k—Z]_l—[(Ar^4)_o—W]_p—$  wherein  $Ar^1$ ,  $Ar^2$ ,  $Ar^3$ , and  $Ar^4$  are the same or different and are arylene units of 6 to 18 carbon atoms; W, X, Y, and Z are the same or different and are bivalent linking groups selected from  $—SO_2—$ ,  $—S—$ ,  $—SO—$ ,  $—CO—$ ,  $—O—$ ,  $—COO—$  or alkylene or alkylidene groups of 1 to 6 carbon atoms and wherein at least one of the linking groups is  $—S—$ ; and n, m, i, j, k, l, o, and p are independently zero or 1, 2, 3, or 4, subject to the proviso that their sum total is not less than 2. The arylene units  $Ar^1$ ,  $Ar^2$ ,  $Ar^3$ , and  $Ar^4$  may be selectively substituted or unsubstituted. Advantageous arylene systems are

phenylene, biphenylene, naphthylene, anthracene and phenanthrene. The polyarylene sulfide typically includes at least 30 mol %, particularly at least 50 mol % and more particularly at least 70 mol % aryene sulfide ( $\text{—S—}$ ) units. Preferably the polyarylene sulfide polymer includes at least 85 mol % sulfide linkages attached directly to two aromatic rings. Advantageously the polyarylene sulfide polymer is polyphenylene sulfide (PPS), defined herein as containing the phenylene sulfide structure  $\text{—(C}_6\text{H}_4\text{—S)}_n\text{—}$  (wherein  $n$  is an integer of 1 or more) as a component thereof.

At least one other of the polymeric components includes polymethylpentene. While mixtures of the polymers may be used, the at least one other polymeric component does not include a polyarylene sulfide polymer as defined above.

The invention is further directed to a method for increasing the acid resistance of any of the embodiments of a polyarylene fiber described herein by providing it with a coating of the second component in any of the embodiments described herein.

In particular the method for improving the acid resistance of a fiber comprises the steps of;

- i. providing a fiber,
- ii. coating the fiber with a thermoplastic polymer that is free of polyarylene sulfide polymer to form a coated fiber, wherein said thermoplastic polymer forms the entire exposed surface of the coated fiber and consists essentially of polymethyl pentene,

said fiber comprising at least polyarylene sulfide polymer.

## **EXAMPLES**

### **Masterbatch**

A PPS composition containing 11.0 weight percent Zinc Octoate was produced using an extrusion process. Fortron ®0309 PPS (89 parts) was melt compounded in a Coperion 18mm intermeshing co-rotating twin-screw extruder with a liquid metering pump adding Zinc Octoate (11 parts) down stream into the melted polymer. The conditions of extrusion included a maximum barrel temperature of 300 °C, a maximum melt temperature of 310 °C, screw speed of 300 rpm, with a residence time of approximately 1 minute and a die pressure of 14-15 psi at a single strand die. The strand was frozen in a 6 ft tap water trough prior to being pelletized to give a pellet count of 100-120 pellets per gram.

### **Spinning Experiment**

In general, polymers are made into fibers by melting the polymer and pushing this viscous fluid through several small orifices as a collection to produce a multifiber yarn. The diameter of the fibers, usually expressed as denier which is the weight of 9000 meters of fiber [or yarn], is established by how fast the polymer is feed through the orifices and how fast this collection is pulled away from the orifices. This pulling with the diameter reduction step mostly occurs where this viscous polymer fluid has cooled sufficiently to again become solid. The pulling is accomplished by wrapping the solid fibers around a rotating roll several times, where either a non-driven roll, aka idler roll, or a second roll driven at the same speed are used in tandem; to permit the several turns or wrappings of the fiber 'threadline' to be spaced from each other the two rolls are canted with regard to each other. This prevents threadline cross overs which can breakdown a continuous removal of the fibers to a next set of rolls for further processing. The fibers are usually wrapped several times around rolls to produce sufficient drag or resisting friction that the fibers maintain the roll speeds without slipping. The fiber diameters may be further reduced by a 'drawing' step, where yarns are drawn, aka extended in length, from the one roll (or a pair) rotating at one speed to another (or pair) moving at a higher speed. This would be a single stage draw.

When the draw process is repeated more than once with additional rolls, this is a multistage draw. A draw assist device may be used between pairs of rolls such as a heated pin or plate, or a hot gas jet which impinges on the yarn. Rolls can also serve other functions such as forwarding the fibers from one position to another. In undrawn fibers such as partially oriented yarns (POY), the rolls serve the purpose to bring the fibers to a speed that will match their winding speed where the fibers are collected on bobbins. Frequently the winding speed will be slightly less than the feeding speed to keep winding tension sufficiently low that fibers do not relax some of their elasticity on the packaged bobbin, and give a poorly formed package. This tension adjustment is also a consideration with drawn fibers.

With drawn fibers, the draw process might benefit fiber properties, or the process, if they are heated, or from heating after the draw on additional rolls as an 'annealing' step. Semi-crystalline polymers, as opposed to amorphous, develop crystallinity in the draw and annealing steps. In general, higher crystallinity gives lower shrinkage, frequently an essential property for fibers. While the temperature of the rolls is sometimes used as a drawing assist, roll temperature can impact final crystallinity and shrinkage. Without annealing, small amounts of fibers can be wound on bobbins without detriment where an elastic recovery hasn't built sufficient force to affect bobbin quality, which might occur on bigger bobbins, i.e. more fiber length on the bobbin. After the draw step additional rolls, if used, will generally spin at slower speeds to let down the elasticity in the fibers with or without heat.

When heated for fiber annealing, an increase in crystallinity at this stage also causes the fibers to want to shrink, and roll speeds are usually lowered in speed to accommodate the tension which is developed from fiber shrinkage. Annealed fibers have less final shrinkage and also have less elasticity memory when transferred to the bobbin which can give better large bobbins. Historically, this is called a continuous filament process.

### **Comparative Fiber Example 1**

In this example, a fiber was made from polyphenylene sulfide component. The resin is available from Ticona as Fortran PPS 309. Before fibers were spun

the resin was dried for 16 hours at 100°C in a vacuum oven with a dry nitrogen sweep. The dried polymer pellets were metered into a Werner and Pfleiderer 28mm twin screw extruder and spun through a 34-hole spinneret orifice of 0.012 inch (0.030 mm) diameter and 0.048 inch (1.22 mm) length. The extruder was heated in the feed zone to 190°C then to melt zones at 275 then 285°C, then transfer zones at 285°C and then to Zenith pumps (available Zenith Pumps, Monroe, NC) at 285°C and then pushed and transferred to the spinneret pack block at 290°C. A ring heater was used at 290°C around the pack nut that holds the spinneret. After simple cross flow air quenching, the undrawn yarns were processed as described below. The wind up unit was a Barmag SW 6.

The speed of the gear pump on the sheath side was preset so as to supply 32.8 g/min of the PPS to the spinneret. The polymer stream was filtered through three 200 mesh screens sandwiched between 50 mesh screens within the pack, and after filtration, a total of 34 individual fibers/ filaments were created at the spinneret orifice outlets with the sheath-core cross section. These 34 resulting filaments were cooled in ambient air quench zone, given an aqueous oil emulsion (10% oil) finish, and then combined in a guide approximately eight feet (~7 meters) below the spin pack. The 34 filament yarn was pulled away from the spinneret orifices and through the guide by a roll with an idler roll turning at approximately 527 meters/minute. From these rolls the yarn was taken to a pair of rolls also at 537 meters/minute, then through a steam jet at 170C, then to a pair of rolls at 1900 meters/minute heated at 125°C, then to a pair of rolls at 1900 meters/minute at room temperature then to a pair of let down rolls and to the windup. The denier on this fiber was 110.

### **Comparative Fiber Example 2**

In this example, a fiber was made from polyphenylene sulfide component with a stabilizer Zinc Octoate. The resin is available from Ticona as Fortran PPS 309.. Before fibers were spun the PPS resin and Masterbatch A was dried for 16 hours at 100°C in a vacuum oven with a dry nitrogen sweep. A combination of

dried polymer pellets in the ratio of (80 parts PPS 309 and 20 parts Masterbatch A) were metered into a Werner and Pfleiderer 28mm twin screw extruder and spun through a 34-hole spinneret orifice of 0.012 inch (0.030 mm) diameter and 0.048 inch (1.22 mm) length. The extruder was heated in the feed zone to 190°C then to melt zones at 275 then 285°C, then transfer zones at 285°C and then to Zenith pumps (available Zenith Pumps, Monroe, NC) at 285°C and then pushed and transferred to the spinneret pack block at 290°C. A ring heater was used at 290°C around the pack nut that holds the spinneret. After simple cross flow air quenching, the undrawn yarns were processed as described below. The wind up unit was a Barmag SW 6.

The speed of the gear pump on the sheath side was preset so as to supply 32.8 g/min of the PPS to the spinneret. The polymer stream was filtered through three 200 mesh screens sandwiched between 50 mesh screens within the pack, and after filtration, a total of 34 individual fibers/ filaments were created at the spinneret orifice outlets with the sheath-core cross section. These 34 resulting filaments were cooled in ambient air quench zone, given an aqueous oil emulsion (10% oil) finish, and then combined in a guide approximately eight feet (~7 meters) below the spin pack. The 34 filament yarn was pulled away from the spinneret orifices and through the guide by a roll with an idler roll turning at approximately 527 meters/minute. From these rolls the yarn was taken to a pair of rolls also at 537 meters/minute, then through a steam jet at 170C, then to a pair of rolls at 1900 meters/minute heated at 125°C, then to a pair of rolls at 1900 meters/minute at room temperature then to a pair of let down rolls and to the windup. The denier on this fiber was 115.

### **Fiber Example A**

In this example, a bicomponent fiber was made from polyphenylene sulfide component as the core and polymethyl pentene as the sheath. The polyphenylene sulfide (PPS) resin is available from Ticona as Fortran PPS 309. The polymethyl pentene (PMP) resin is available from Mitsui Chemicals America

as DX820. Before fibers were spun the resin was dried for 16 hours at 100°C in a vacuum oven with a dry nitrogen sweep. The dried polymer pellets were metered into two separate Werner and Pfleiderer 28mm twin screw extruder (one for the core and the other for the sheath) and spun through a 34-hole spinneret orifice of 0.012 inch (0.030 mm) diameter and 0.048 inch (1.22 mm) length. The extruder feeding the sheath side containing polymethyl pentene was heated in the feed zone to 200°C then to melt zones at 245 then 275°C, then transfer zones at 275°C and then to Zenith pumps (available Zenith Pumps, Monroe, NC) at 275°C and then pushed and transferred to the spinneret pack block at 290°C. The extruder feeding the core section containing polyphenylene sulfide was heated in the feed zone to 190°C then to melt zones at 275 then 285°C, then transfer zones at 285°C and then to Zenith pumps (available Zenith Pumps, Monroe, NC) at 285°C and then pushed and transferred to the spinneret pack block at 290°C. A ring heater was used at 290°C around the pack nut that holds the spinneret. After simple cross flow air quenching, the undrawn yarns were processed as described below. The wind up unit was a Barmag SW 6.

The speed of the gear pump on the sheath side was preset so as to supply required amount of PMP while the gear pump on the core side was preset to required amount of the PPS to the spinneret. The polymer stream was filtered through three 200 mesh screens sandwiched between 50 mesh screens within the pack, and after filtration, a total of 34 individual fibers/ filaments were created at the spinneret orifice outlets with the sheath-core cross section. These 34 resulting filaments were cooled in ambient air quench zone, given an aqueous oil emulsion (10% oil) finish, and then combined in a guide approximately eight feet (~7 meters) below the spin pack. The 34 filament yarn was pulled away from the spinneret orifices and through the guide by a roll with an idler roll turning at approximately 540 meters/minute. From these rolls the yarn was taken to a pair of rolls also at 540 meters/minute, then through a steam jet at 170C, then to a pair of rolls at 1800 meters/minute heated at 125°C, then to a pair of rolls at 1800 meters/minute at room temperature then to a pair of let down rolls and to the windup.

Example #		Composition (weight % based on the total weight of the fiber)		Basis weight (denier)
		Sheath	Core	
A	A-1	30% PMP	70% PPS	122
	A-2	25% PMP	75% PPS	121
	A-3	20% PMP	80% PPS	122

### Fiber Example B

In this example, a bicomponent fiber was made from polyphenylene sulfide containing zinc ethylhexanoate stabilizer component as the core and polymethyl pentene (PMP) as the sheath. Before fibers were spun the PMP, PPS and Masterbatch A resin were dried for 16 hours at 100°C in a vacuum oven with a dry nitrogen sweep.

The dried polymer pellets were metered into two separate Werner and Pfleiderer 28mm twin screw extruder (one for the core and the other for the sheath) and spun through a 34-hole spinneret orifice of 0.012 inch (0.030 mm) diameter and 0.048 inch (1.22 mm) length. The extruder feeding the sheath side containing polymethyl pentene was heated in the feed zone to 200°C then to melt zones at 245 then 275°C, then transfer zones at 275°C and then to Zenith pumps (available Zenith Pumps, Monroe, NC) at 275°C and then pushed and transferred to the spinneret pack block at 290°C.

A blend of PPS resin (80 wt%) and Masterbatch A (20 wt%) was fed into the extruder feeding the core section. This extruder was heated in the feed zone to 190°C then to melt zones at 275 then 285°C, then transfer zones at 285°C and then to Zenith pumps (available Zenith Pumps, Monroe, NC) at 285°C and then pushed and transferred to the spinneret pack block at 290°C. A ring heater was used at 290°C around the pack nut that holds the spinneret. After simple cross flow air quenching, the undrawn yarns were processed as described below. The wind up unit was a Barmag SW 6.

The speed of the gear pump on the sheath side was preset so as to supply required amount of PMP while the gear pump on the core side was preset to required amount of the PPS to the spinneret. The polymer stream was filtered through three 200 mesh screens sandwiched between 50 mesh screens within the pack, and after filtration, a total of 34 individual fibers/ filaments were created at the spinneret orifice outlets with the sheath-core cross section. These 34 resulting filaments were cooled in ambient air quench zone, given an aqueous oil emulsion (10% oil) finish, and then combined in a guide approximately eight feet (~7 meters) below the spin pack. The 34 filament yarn was pulled away from the spinneret orifices and through the guide by a roll with an idler roll turning at approximately 540 meters/minute. From these rolls the yarn was taken to a pair of rolls also at 540 meters/minute, then through a steam jet at 170C, then to a pair of rolls at 1800 meters/minute heated at 125°C, then to a pair of rolls at 1800 meters/minute at room temperature then to a pair of let down rolls and to the windup.

Example #		Composition (weight % based on the total weight of the fiber)		Basis weight (denier)
		Sheath	Core	
B	B-1	30% PMP	70% PPS (2.2% Zinc Octoate)	122
	B-2	25% PMP	75% PPS (2.2% Zinc Octoate)	121
	B-3	20% PMP	80% PPS (2.2% Zinc Octoate)	122

### Acid Test Experiment on the Fibers

A bicomponent fiber, approximately 2 meter in length, prepared by the above mentioned process is wound on glass rod. The glass rod with the fiber is placed in a vial containing an acid mixture. The acid mixture is made up of 10:40:50 wt% of nitric acid (70% concentrated), sulfuric acid (98% concentrated) and distilled water respectively. Care is taken to ensure that the fibers are not in direct contact with the acid solution. The vial is sealed with a cap once the glass rod with the fiber is placed inside it. The sealed vial containing the fiber is placed in a mantle with slots for the vials and heated to 120°C. The vials with the fiber samples are removed for testing at an interval of two, four and six hours. The fibers are then rinsed with water several times, dried in air overnight and unwound carefully. The unwound fibers are then tested for tenacity and elongation.

Table 1 summarizes the sample types.

Table 1

Sample	Core	Sheath
Comp 1	PPS	NA
Comp 2	PPS (2.2% Zinc Octoate)	NA
A-1	70% PPS	30% PMP
A-2	75% PPS	25% PMP
A-3	80% PPS	20% PMP
B-1	70% PPS (2.2% Zinc Octoate)	30% PMP
B-2	75% PPS (2.2% Zinc Octoate)	25% PMP
B-3	80% PPS (2.2% Zinc Octoate)	20% PMP

The results of tenacity and elongation testing on these treated and untreated fibers are given in the table below. Tenacity and elongation of the fibers were measured on an Instron-type testing machine with a gage length of 10 cm, test speed of 6 inch/min in accordance with ASTM D2256.

Table 2

Tenacity and tenacity retention of the fibers treated with the acid mixture at 0, 2, 4 and 6hrs

Sample ID	Time							
	0	hrs	2	hrs	4	hrs	6	hrs
	tenacity (g/den)	tenacity retention (%)	tenacity (g/den)	tenacity retention (%)	tenacity (g/den)	tenacity retention (%)	tenacity (gpf)	tenacity retention (%)
Comp 1	3.24	100	2.49	76.9	2.46	75.9	1.98	61.1
Comp 2	3.17	100	2.63	82.9	2.33	73.5	1.85	58.4
A-1	3.58	100	3.73	104.2	3.63	101.4	3.42	95.5
A-2	3.68	100	3.4	92.4	3.15	85.6	2.96	80.4
A-3	3.8	100	3.2	83.9	3.4	89.5	3.34	87.9
B-1	3.41	100	3.34	97.9	3.15	92.4	3.04	87.1
B-2	3.51	100	3.32	94.6	3.04	86.6	2.72	77.5
B-3	3.56	100	3.28	92.1	3.07	86.2	3.12	87.6

Table 3

Elongation and elongation retention of the fibers treated with the acid mixture at 0, 2, 4 and 6hrs

Sample ID	0 hrs		2 hrs		4 hrs		6 hrs	
	elongation (%)	elongation retention (%)	elongation (%)	elongation retention (%)	elongation (%)	elongation retention (%)	elongation (%)	elongation retention (%)
Comp 1	24.9	100	18.8	75.5	16.2	64.8	11.03	44.2
Comp 2	25.4	100	19.4	76.3	15.1	59.4	11.52	45.3
A-1	25.6	100	25.6	99.9	26.4	103.2	24.1	94.3
A-2	24.8	100	22.7	91.6	21.5	86.8	18.92	76.5
A-3	28.1	100	21.4	72.6	15.4	76.1	22.05	78.4
B-1	23.1	100	23.5	101.5	20.6	88.9	19.7	85.0
B-2	25.2	100	22.6	89.7	20.0	79.5	17.21	68.3
B-3	26.4	100	22.2	84.1	19.6	74.4	21.08	79.9

Tables 2 and 3 show the ability of the fibers of the invention to resist an acid environment at the temperature of the test. Tenacity retention in the absence of the PMP coating after 6 hours is around 60%, while in the coated samples it goes up to around 80%. A similar trend is seen with elongation.

We claim;

1. A multicomponent fiber having an exposed outer surface, said fiber comprising: at least a first component of polyarylene sulfide polymer; and at least a second component of a thermoplastic polymer free of polyarylene sulfide polymer, wherein said thermoplastic polymer forms the entire exposed surface of the multicomponent fiber and consists essentially of polymethylpentene.
2. The fiber of claim 1, wherein said polyarylene sulfide polymer comprises a polymer in which at least 85 mol % of the sulfide linkages are attached directly to two aromatic rings.
3. The fiber of claim 2, wherein said polyarylene sulfide polymer is polyphenylene sulfide.
4. The fiber of claim 1, wherein said second component is present at a 10 to 30% by weight of the total polyarylene sulfide plus thermoplastic polymer.
5. The fiber of claim 1, wherein the second component comprises less than about 30 percent by weight of the total weight of the fiber.
6. The fiber of claim 5, wherein the second component comprises less than about 20 percent by weight of the total weight of the fiber.
7. The fiber of claim 1, wherein said fiber has a circular cross section.
8. The fiber of claim 1, wherein said fiber has a multi-lobal cross section.

9. The fiber of claim 1, wherein said fiber is a continuous filament.
10. The fiber of claim 1, wherein said fiber is a staple fiber.
11. The fiber of claim 1, wherein said fiber is a spunbond fiber.
12. The fiber of claim 1, wherein said fiber is a meltblown fiber.
13. The fiber of claim 1, wherein said fiber is a bicomponent fiber comprising a sheath component and a core component, wherein said sheath component forms the entire exposed outer surface of said fiber and comprises said thermoplastic polymer free of polyarylene sulfide polymer, and wherein said core component comprises polyarylene sulfide polymer.
14. The fiber of claim 22, wherein said bicomponent fiber has a concentric sheath/core cross section.
15. The fiber of claim 22, wherein said bicomponent fiber has an eccentric sheath/core cross section.
16. The fiber of claim 1, wherein said fiber is an islands-in-the-sea fiber comprising a sea component and a plurality of island components distributed within said sea component, wherein said sea component forms the entire exposed outer surface of said fiber and comprises said thermoplastic polymer free of polyarylene sulfide polymer, and wherein said plurality of island components comprises polyarylene sulfide polymer.

17. A web comprising the fiber of claim 1.
18. The web of claim 17, wherein the web comprises a woven or nonwoven material.
19. The web of claim 18, wherein the web is made from a spunbond or meltblown process.
20. A method for improving the acid resistance of a fiber, comprising the steps of;
- providing a fiber,
- coating the fiber with a thermoplastic polymer that is free of polyarylene sulfide polymer to form a coated fiber, wherein said thermoplastic polymer forms the entire exposed surface of the coated fiber and consists essentially of polymethylpentene, and
- said fiber comprises at least a first component of polyarylene sulfide polymer.

FIG. 1

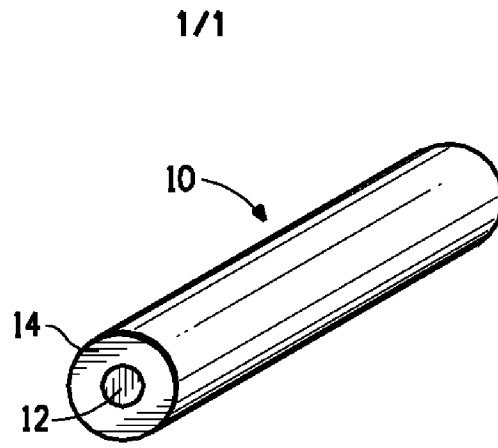


FIG. 2

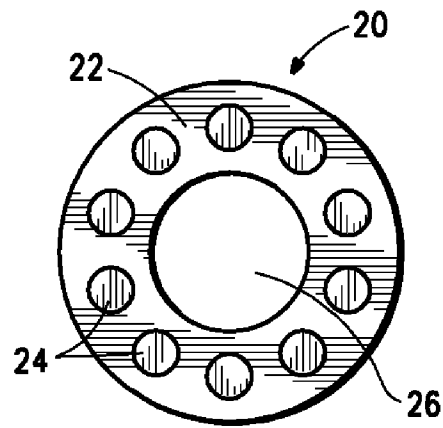
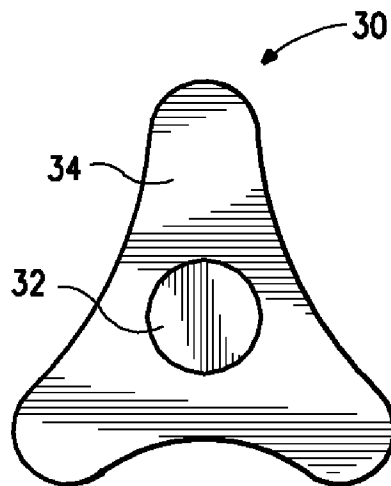


FIG. 3



# INTERNATIONAL SEARCH REPORT

International application No <b>PCT/US2014/033246</b>
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**A. CLASSIFICATION OF SUBJECT MATTER**  
 INV. D01F8/16            D01F8/06  
 ADD. B01D39/16        D01D5/30

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)  
 D01F D01D B01D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2009/076459 A1 (DU PONT [US]; ROLLIN JR PAUL ELLIS [US]; MA XUN [US]; YOST BRUCE A [US] 18 June 2009 (2009-06-18) abstract; claims 1-3; figures 1-3 page 7, lines 10-13 the whole document	1-20
A	----- WO 00/42244 A1 (SOLUTION INC [US]) 20 July 2000 (2000-07-20) page 8, lines 15-26; claims 1,2,16; figure 1	1-20
A	----- JP H02 127521 A (DAIWA SPINNING CO LTD) 16 May 1990 (1990-05-16) abstract; figure 1 -----	1-20

Further documents are listed in the continuation of Box C.

See patent family annex.

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Date of the actual completion of the international search

Date of mailing of the international search report

16 July 2014

24/07/2014

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/US2014/033246

Patent document cited in search report	Publication date	Patent family member(s)	Publication date	
WO 2009076459	A1	18-06-2009	CN 101896652 A	24-11-2010
			EP 2220272 A1	25-08-2010
			JP 2011506790 A	03-03-2011
			US 2009156075 A1	18-06-2009
			WO 2009076459 A1	18-06-2009
-----				
WO 0042244	A1	20-07-2000	AU 2213900 A	01-08-2000
			WO 0042244 A1	20-07-2000
-----				
JP H02127521	A	16-05-1990	NONE	
-----				